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Adsorption of Mercuric Chloride from Solution by Gladiolus Corms

By R. H. Nelson, *assistant entomologist, Division of Truck Crop and Garden Insect Investigations,* and C. C. Cassil, *associate chemist, Division of Insecticide Investigations, Bureau of Entomology and Plant Quarantine*

CONTENTS

INTRODUCTION

Mercuric chloride, also called bichloride of mercury and corrosive sublimate, is a well-known poisonous compound. In aqueous solutions of various strengths it is used for a number of purposes in addition to its horticultural application as a fungicide and insecticide.

The studies herein reported were undertaken to determine by precise chemical analysis just how much mercuric chloride is removed from solution by unpeeled gladiolus corms when they are soaked for various periods of time. It was hoped that the data would give some information on the possible reuse of a solution.

REVIEW OF LITERATURE

Previous to the advent in the commercial fields of the gladiolus thrips (*Taeniothrips simplex* Mor.) solutions of mercuric chloride, usually 1 to 1,000, were used by gladiolus growers as a treatment for certain corm-borne diseases. Shortly after this thrips was found to feed upon the stored corms, Dustan,[1] of the Canadian Department of Agriculture, suggested that the corrosive sublimate treatment could also be used against the thrips. In a subsequent publication [2] he recommended at 70° F. a solution of 1 ounce of the chemical in 6

[1] Dustan, Alan G. the gladiolus thrips and its control. Canada Dept. Agr. D. F. C. and G. I. No. 101, Ottawa 34, 5 pp. 1931. (Mimeographed.)

[2] —— recommendations for the control of the gladiolus thrips in 1932. Ibid. 102, Ottawa 35, 5 pp. 1932. (Mimeographed.)

gallons (Imperial) of water, a dilution of 1 to 960 or 0.104 percent, in which the corms with the tunic removed were to be soaked for 3 hours. This recommendation is still in effect in Canada. In the case of unpeeled corms a 20-hour soak is advised (*2*).[3]

Weigel, Smith, and Richardson (*12*) recommended a solution of 1 ounce in 8 gallons of water, with an immersion period of 3 hours. They stated that this was a 1 to 1,000 solution. Actually it is 1 to 1,067 (0.0937 percent). Subsequently Smith and Richardson (*8*) found the 3-hour immersion period to be not completely effective on unpeeled corms. They obtained complete control in one test with an 8-hour soak but indicated that they had reports of longer immersions proving ineffective. Weigel (*11*) recommended soaking the corms for 17 hours in the solution of 1 ounce in 8 gallons. Herr (*3*) found 1 ounce in 7½ gallons, with a 4-hour soak, to be effective. Tate and Poor (*10*) suggest the solution of 1 ounce to 8 gallons, with an immersion period of 8 to 10 hours for unpeeled corms and 3 to 4 hours for peeled corms.

In none of the publications to which reference has just been made is there mention of methods for maintaining the strength of the solution when it is used for more than one loading of corms, as is often done by commercial growers. List (*5*) recommends a solution of 1 ounce of the mercuric chloride in 7½ gallons of water, 1 to 1,000, or 0.1 percent, in which peeled corms are to be soaked 4 hours and unpeeled corms 8 to 12 hours. He states that the solution may be used again if it is brought back to the original level and one-third of the initial quantity of mercuric chloride is added. This recommendation for maintaining the strength of the solution appears repeatedly in the literature on gladiolus culture and diseases.

Knowlton (*4*) recommended that the corms be dipped 12 to 18 hours in a 1-ounce-to-8-gallon solution and that the solution be used only once. McKenzie (*6*) recommended a solution of 1 ounce in 7 gallons of water (1 to 934, or 0.107 percent) with a soak of 17 hours for unpeeled corms. For corms with the tunic removed he suggested a solution of 1 ounce in 6 gallons (1 to 800, or 0.125 percent) with an immersion period of 3 hours. He stated that more of the stock solution must be added after each treatment.

As is evident from the foregoing brief review of the literature, there is no general agreement on the exact strength of the solution to be used, the duration of immersion, the possibility of the reuse of a solution, or how the strength of the solution may be maintained if it is reused.

A chemical method for testing the strength of mercuric chloride solutions when used for treating potatoes has been recommended by the New York (Cornell) Agricultural Experiment Station (*7*). This test can be used in connection with the soaking of gladiolus corms as well as for potatoes. Essentially, the test consists of titrating a sample of the freshly prepared mercuric chloride solution into a measured quantity of a 0.5-percent solution of potassium iodide, the end point being the appearance of mercuric iodide as a red precipitate. The quantity of the mercuric chloride solution used for the titration is recorded. In the case of potatoes, two lots are treated and the solution is then returned to the original volume with water and retested the same as before, using the same quantity of potassium

[3] Italic numbers in parentheses refer to Literature Cited, p. 9.

iodide solution. The increased quantity of the corrosive sublimate solution required, as compared with that used for the freshly prepared solution, to reach the end point in titration indicates how much the dip has been weakened. Given the amount of mercuric chloride in the original solution, the amount required to bring it back to the desired strength can then be calculated.

METHOD OF TREATMENT

Numerous tests by the senior author, in which a 1 to 1,000, or 0.1-percent, solution have been used, have consistently resulted in a complete kill of all stages of the gladiolus thrips when the unpeeled corms were soaked for from 12 to 17 hours. To provide a margin of safety, the 17-hour, or overnight, immersion period is recommended.

All initial solutions were made up at the rate of 1 gram of mercuric chloride per liter of water, that is, 1 to 1,000, or a 0.1-percent solution. The chemical was first dissolved in a small quantity of hot water and then diluted to the desired strength. Earthenware and wooden containers were used.

Corms of the variety and size hereinafter detailed for each test were then weighed out. Weight rather than the number of corms was used in most tests so that the loads would be as nearly equal as possible for the replicates in each test. Sufficient corms were used in each lot so that there would be just enough solution to cover them, along with the burlap sack in which they were placed—that is, a capacity load. The use of burlap sacks and capacity loading simulate commercial practices. Immersion periods of 2, 7, 12, and 17 hours were used, and in one test with burlap sacks alone a soaking of 24 hours was given. At the end of the soaking period the sacked corms were removed and allowed to drain back into the container for a few minutes. After the corms had drained, the liquid in the container was thoroughly stirred and then sampled for chemical analysis.

The temperature of the solution was taken at the beginning and end of each test. In most of the tests the temperature did not change during the treating period, as the work was done in the corm-storage room where the air temperature did not vary to any great extent.

Analytical Method

The method used for analyzing the corm tunic and tissue was essentially that prescribed by the Association of Official Agricultural Chemists (*1, pp. 393–396*).

In brief, the tunic or tissue was partly digested with nitric acid, filtered, a portion completely digested with potassium permanganate, and the manganese dioxide destroyed with hydrogen peroxide. The resulting solution was then titrated with a carbon tetrachloride solution of diphenylthiocarbazone.

It was found by experiment that the organic material extracted from the corms by water did not interfere with the mercury titration. Therefore, the concentration of mercuric chloride in the original and used solutions was determined by a direct titration of a 5–10 milliliter sample with diphenylthiocarbazone.

Two-Hour Tests

An immersion period of 2 hours is not sufficient to effect a complete kill of the gladiolus thrips, but this length of soaking was included so that comparison could be made between the quantities of chemical removed during this and the longer periods.

The corms used were mixed sizes Nos. 1 and 2 of the variety Alice Tiplady. After the first test, samples were taken and the unaltered solution was reloaded for a second run and also for a third. The decreased quantity of corms used from test to test was necessitated by the removal of the sample and the absorption of solution by the previous lot. The results of these tests are presented in table 1, tests 1, 2, and 3.

TABLE 1.—*Adsorption of mercuric chloride from solution by several varieties of gladiolus corms, Beltsville, Md., 1937*

Test No.[1]	Quantity of corms per lot	Replicate lots	Immersion period	Temperature of solution	Mean initial concentration of mercuric chloride per liter	Mean final concentration of mercuric chloride per liter	Mean mercuric chloride removed per liter
	Grams	*Number*	*Hours*	*°F.*	*Grams*	*Grams*	*Grams*
1	2,725	4	2	60	1.0	0.68	0.32
2	2,270	4	2	60	.68	.36	
3	1,815	4	2	60	.36	.22	
4	2,725	6	7	60	1.0	.48	.52
5	2,270	6	7	60	.48	.24	
6	1,815	6	7	60	.24	.09	
7	2,725	6	12	68–70	1.0	.42	.58
8	2,270	6	12	68–70	.42	.097	
9	1,815	6	12	68–70	.097	.041	
10	5,443	6	17	60	1.0	.45	.55
11	2,725	6	17	73–75	1.0	.34	.66

[1] Required differences for significance for odds of 19 to 1:

Between mean of test 1 and any 1 of means of tests 4, 7 or 10	0.053
Between any 2 means of tests 4, 7, and 10	.048
Between means of tests 10 and 11	.052

Seven-Hour Tests

A soaking period of 7 hours is likewise not completely effective against the gladiolus thrips, but was included for the same reasons as previously explained for the 2-hour immersions.

The varieties used for each test were Joe Coleman and mixed varieties of *Gladiolus primulinus*, there being four replicates of the former and two replicates of the latter. The corms were mixed sizes Nos. 1 and 2. Otherwise the procedure was the same as for the 2-hour tests (table 1, tests 4, 5, and 6).

Twelve-Hour Tests

As previously stated, a dipping period of 12 hours has been found effective against the gladiolus thrips.

Four lots of the variety Brilliant and two of Cardinal Prince, mixed sizes Nos. 1 and 2, were used in each test. The procedure was the same as for the tests of shorter duration (table 1, tests 7, 8, and 9).

All recommendations known to the authors for the use of this material as a thripicide specify solutions of or near 1 to 1,000. The

writers have no data and know of none published which suggest that concentrations such as are shown in table 1 following the initial soakings of 7, 12, or 17 hours (less than 1 to 2,000) would be effective against the gladiolus thrips. In view of the length of soaking necessary to obtain a complete kill with the 1 to 1,000 solution, it is indeed doubtful whether concentrations of approximately one-half this strength would result in complete thrips mortality even with a rather long immersion period. Reuse of the unaltered solution, therefore, appears out of the question and was not tested further.

Seventeen-Hour Tests

Seventeen hours is the recommended immersion period for a complete kill of the gladiolus thrips on the corms.

Two lots each of the varieties Crimson Glow, Alice Tiplady, and of mixed varieties of *Gladiolus primulinus* were used in the test (table 1, test 10). No tests were made on the reuse of the solution. Larger quantities of corms per lot, with consequent porportionate increase in the volume of the initial solution, were used than in the preceding tests.

In this 17-hour experiment measurements were also made of the reduction in the volume of the solution for each of the six replicates at the end of the immersion period. These reductions ranged from 7.5 to 12.5 percent, with an average of 10.8. This loss in volume is small when contrasted with the 55-percent reduction in the concentration of the chemical in the solution.

COMPARISONS ON THE BASIS OF THE IMMERSION PERIOD

Tests 1, 4, 7, and 10, as summarized in table 1, show the effect of the corms at the four different immersion periods on the initial or 1-gram-per-liter solutions. The data from which the means of these four tests were obtained were analyzed for variance as outlined by Snedecor (*9*), and the results are shown in table 1. The differences between the mean quantity of chemical removed by the 2-hour immersion and that removed by any one of the three longer soakings are significant. Adsorption appears to be definitely increased by the longer immersion periods. There is no real difference between the 7- and 17-hour immersions or between the 12- and 17-hour soakings in the quantity of mercuric chloride removed from solution. However, the difference between the 7- and 12-hour means is slightly greater than that required for significance. The reason for this is, perhaps, the fact that in the 12-hour immersion tests the solution temperature was 68°–70° F., whereas it was 60° for the 7- and 17-hour soakings. In any event it seems evident that at the solution temperature of 60° a 17-hour immersion period does not increase the mercuric chloride adsorption over that of a 7-hour soaking. This is of interest when considered in regard to thrips mortality. Tests which were made previous to this work showed a 94.6-percent kill with a 7-hour soaking and a 100-percent kill with 12-hour and 17-hour soakings. The surviving thrips in the 7-hour tests were found on only a small percentage of the total corms treated, and on these only in certain

small areas well protected by the scales. Since no additional chemical is taken up after 7 hours, the complete kill which follows the longer soakings must be due simply to the fact that the immersion period is sufficiently long to permit the solution to penetrate to these more protected areas. Apparently the small additional surface reached in the longer soakings is not enough to increase the adsorption significantly.

EFFECT OF TEMPERATURE

Some further light on the effect of solution temperature on the quantity of chemical adsorbed by the corms is given by the data for the two 17-hour tests summarized in table 1. Test 11 was run with corms of the variety Cardinal Prince, mixed sizes Nos. 1 and 2.

The data show that at a solution temperature of 73°–75° F. more mercuric chloride was removed from solution than at 60°, and that the difference between the quantities removed is highly significant. This difference is presumably due to the temperatures of the solutions, although it may be due in part to varietal effect.

CORM-SIZE TESTS

The tests summarized in table 2 were made to determine the difference, if any, in adsorption due to surface effect of large and small corms. The same quantity of solution and approximately the same volume of corms were used in each test. The corms for test 12 were 2 to 3 inches in diameter (size No. 1 Jumbo), while those in test 13 were 1 to 1¼ inches across (size No. 3). The grading was carefully done so that the weights of the various lots in each test would be as nearly equal as possible while at the same time, each lot would be composed of a definite number of corms. The variety Cardinal Prince was used in both tests.

TABLE 2.—*Adsorption of mercuric chloride from solution by Cardinal Prince gladiolus corms of two sizes during a 7-hour soaking period at a solution temperature of 66–68° F., Beltsville, Md., 1937*

[The initial concentration of mercuric chloride per liter was 1 gram in each case]

Test No.	Corms per lot	Weight of corms	Final concentration of mercuric chloride per liter	Mercuric chloride adsorbed [1] by—		Mercuric chloride per liter of initial solution recovered
				Corm tissue	Tunic	
	Number	*Grams*	*Grams*	*Grams*	*Grams*	*Grams*
12	50	1,900	0.51	0.043	0.188	0.741
	50	1,885	.49	.020	.196	.706
	50	1,900	.53	.043	.157	.730
Mean			.51	.035	.180	.725
13	200	1,875	.46	.048	.184	.692
	200	1,870	.50	.053	.222	.775
	200	1,875	.48	.045	.212	.737
Mean			.48	.049	.206	.735
Required difference between means for significance for odds of 19 to 1			.045			.072

[1] Calculated for corms per liter of initial solution from quantities found in 5-corm samples from each lot of test 12, and 10-corm samples from each lot of test 13.

In addition to the analysis of the solution at the end of the treatment, 5 corm samples from each lot of test 12 and 10 from each lot of test 13 were analyzed for mercuric chloride in the tunic and in the corm proper.

The data as presented in table 2 show no significant difference between the mean final concentrations of the solutions for the two tests; that is, corm size made no real difference in the quantity of mercuric chloride adsorbed. From the chemical analysis of the corms themselves it appears that 20 percent or more of the chemical is taken up by the corms, and that on an average the thin scales of the tunic take up approximately 4 to 10 times as much of the chemical as the corm proper for both sizes of corms. When the quantity of mercuric chloride recovered, that is, the final solution concentration plus that found in the corms, is calculated in grams per liter of initial solution and these data are analyzed for variance, the difference between the two means is also not significant. This further indicates that the corm size does not have much effect on adsorption.

BURLAP-SACK IMMERSION TEST

A point of interest in the last column of table 2 is that, on the basis of means, from 26 to 27 percent of the mercuric chloride originally put in is not accounted for. The only apparent explanation was that this percentage had been taken up by the burlap sacks in which the corms were placed for soaking.

A test was made to determine the effect of the sacks alone when soaked for 24 hours in the 1 to 1,000 solution. For each lot a single empty sack such as had been used to hold the corms in previous tests was immersed in 4 liters of solution, which was the quantity used for each lot in tests 12 and 13.

The mean final concentration in the solution of 0.69 gram per liter indicated that under the conditions of the test the sacks removed 0.31 ± 0.037 gram per liter of the mercuric chloride from solution. An adsorption of 31 percent by the sacks in tests 12 and 13 would account almost exactly for the unrecovered chemical noted in the discussion of those tests.

TESTS ON THE RECHARGING OF SOLUTIONS

In the tests on the renewal of the solutions as summarized in table 3 the following procedure was followed: At the end of the 17-hour immersion the corms were removed and a sample was taken as in former tests. Then one-half of the quantity of mercuric chloride present in the initial solution was well stirred in to each container, and another sample taken. With an average reduction of 10 percent in the volume of the solution, this would mean that the chemical was added at the rate of approximately 0.55 gram per liter.

The solutions of test 11 (table 1), where the variety employed was Cardinal Prince, were used as test 11A in these recharging experiments. Two lots each of the gladiolus varieties Brilliant, Cardinal Prince, and Virginia were used for tests 15 and 16. The Brilliant was mixed sizes Nos. 1, 2, and 3, 85 corms per lot; the Cardinal Prince was mixed sizes Nos. 1, 2, and 3 also, but 90 corms per lot; while the Virginia corms were all size No. 1, 90 corms per lot.

TABLE 3.—*Results of recharging mercuric chloride solution by the addition of one-half of the original quantity of the chemical present, after it had been used during a 17-hour soaking period of gladiolus corms of three varieties, Beltsville, Md., 1937–38*

[The mean initial concentration of mercuric chloride per liter was 1 gram in each case]

Test No.[1]	Temperature of solution	Concentration of mercuric chloride per liter after 17-hour soak		Concentration of mercuric chloride per liter in recharged solution			
		Range	Mean	Theoretical[2]		Actual	
				Range	Mean	Range	Mean
	°F.	Grams	Grams	Grams	Grams	Grams	Grams
11A	73–75	0.31–0.38	0.34	0.86–0.93	0.89	0.73–0.78	0.76
15	60	.31– .60	.51	.86–1.15	1.06	.69– .95	.86
16	70	.34– .63	.54	.89–1.18	1.09	.88– .97	.92
Required difference between means for significance for odds of 19 to 1			.125				

[1] 6 replicate lots used in each test.
[2] The 0.55 gram per liter of chemical added plus the concentration at the end of the soaking period.

There are a number of points of interest in the data summarized in table 3. In the first place, considering the mean concentrations at the end of the soaking period, the solution test at 73°–75° F. (11A) was significantly different from that in the 60° and 70° tests but the difference between the last two is not statistically real. The results for tests 11A and 15 check very well with the results presented in table 1. Just why there was not greater adsorption at the 70° temperature than at 60° is not known.

Although the variety Cardinal Prince was common to all three tests, it was used alone in test 11A, but in combination with two other varieties in tests 15 and 16. This detracts somewhat from the conclusiveness of the comparisons.

As regards the recharged solution, it is apparent that two out of the three theoretical mean concentrations are very close to the desired 1-gram-per-liter dilution, the third (test 11A) being below this strength because of the larger quantity of chemical adsorbed from the initial solution. The actual mean concentrations found, however, were all under the desired dilution, ranging from 15 to 19 percent below their respective theoretical means. The authors have no definite explanation for this difference.

When this variation between theoretical and actual concentrations is considered along with the extremes noted for the various lots and the effects of solution temperature on initial adsorption, it becomes evident that the addition to a used solution of one-half the initial quantity of mercuric chloride cannot be relied on to return the solution to the desired 1 to 1,000 strength. Much less reliable are the suggested procedures of returning the solution to the original level and adding one-third of the initial quantity of chemical or of refilling with stock solution.

SUMMARY AND CONCLUSIONS

The data presented in this circular indicate that a capacity load of gladiolus corms contained in a burlap sack soaked for 7 hours or longer removed 50 percent or more of the mercuric chloride from a 1 to 1,000 solution. The length of the soaking period did not significantly affect the quantity adsorbed from solutions of the same temperature. Where 17-hour immersion tests at 60° and at 73°–75° F. were compared, the quantity of chemical removed from solution was significantly increased in the warmer solution.

As contrasted with the chemical removed, the volume of the solution was reduced only about 10 percent by a 17-hour soaking.

It appears that corm size does not affect the quantity of chemical adsorbed, the difference between that taken up by large and small corms under the same conditions being nonsignificant. The data show that about 31 percent of the initial 1 gram per liter of mercuric chloride is taken up by the burlap sacks in an immersion period of 24 hours. The remaining 20 percent or more taken out of the solution is adsorbed by the corms. The thin scales of the tunic adsorbed 4 to 10 times as much mercuric chloride as the corm proper.

The addition of one-half the initial quantity of mercuric chloride to solutions previously used for a 17-hour soaking resulted in a wide variation in the strength of different lots, and none were brought back to the desired concentration.

Under practical conditions, the weight ratio of corms to burlap bags, temperature of the solution, load of corms, and other factors would be subject to considerable variation. In the light of results presented in this circular and considering that an attempt was made to control these variants, it seems evident that no rule-of-thumb method for maintaining the solution at 1 to 1,000 for reuse has been found. It follows, therefore, that gladiolus growers should either prepare a fresh solution for each load of corms in order to have an adequate quantity of corrosive sublimate present, or make use of the potassium iodide titration test previously noted. If this is done, the test and the recharging should be made after each treatment and not after two treatments as recommended for potatoes.

The chemical method used for testing the mercuric chloride solutions in these tests was a titration with a carbon tetrachloride solution of diphenylthiocarbazone. This method is very accurate but is not suited for use by growers since the compound used for titration decomposes easily under various conditions.

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